

# COATINGS

UDC 629.7.023.222

## PROTECTIVE PROCESS COATINGS FOR HOT PLASTIC WORKING OF STEELS AND ALLOYS

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Translated from *Steklo i Keramika*, No. 6, pp. 24–27, June, 2007.

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A technology for dispersion doping of glass matrices for creating superplastic coatings, permitting isothermal deformation of alloys without the use of special equipment, has been developed. The protective glassy and glass-ceramic process coatings which have been developed possess a strong internal structure and high film-forming ability and they withstand substantial impact and tension loads with high strain (above 40%).

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A properly chosen process coating largely predetermines the successful development of a new technology for hot plastic working of metals. Choosing or developing coatings for plastic working is a very difficult problem. It is especially difficult to develop coatings for high-temperature plastic working, since under such conditions the coatings burnup and lose their lubricating properties, and harmful gases are emitted as the coatings burnup. The temperature ranges for thermomechanical working are 900–1200°C for steels and alloys and 1000–2000°C for refractory materials and alloys.

A process coating for hot plastic working of metals must satisfy the following basic criteria:

the separating layer must have a low thermal conductivity;

the corrosion of the surface layer by gases must be prevented;

the adhesion to the metal must be strong and the integrity of the separating layer must be maintained during the deformation of a blank;

the deformation stress of the coating in the separating layer must be low, owing to which the friction coefficient decreases;

the coating must be easy to apply before an article undergoes deformation and easy to remove from the surface of the finished article;

no substances which are harmful to human health should be emitted during plastic working;

any contamination must be relatively small and coating residues must be easy to remove and recoverable.

During hot deformation a heated blank, as a rule, touches a tool which is cooler. Even though contact is of short duration a thin contact layer of the deformed metal cools, and in the process the deformation stress in this region increases by a factor of 2–3. At the same time the contact surface of the tool heats up, and the tool's hardness and strength decrease.

It is known that the heat-insulating layer of a coating with a low thermal conductivity decreases the heat transfer between the metal and the blank and lowers the temperature of the tool's surface.

Commonly used high-temperature coatings are boron nitride, zirconium chloride, lead oxide, alkali-metal sulfides, simple alkali-metal phosphates, and graphite combined with talc.

Any coating used during plastic working of metals must decrease the friction stress and energy consumption. In addition, friction affects the character of the flow of the material and largely determines the quality of the surface of the article. The rubbing conditions affect the stability of the tool. But rubbing and wear are not always interrelated (USSR Inventor's Certificate No. 945765). The friction coefficient depends on the chemical composition of the coating, the viscosity, the microgeometry of the sliding surface, the chemical interaction of the materials and their effect on the conditions of deformation (temperature, force). The thickness of the coating layer at the deformation focus is one of the most important indicators of the rubbing conditions, and this presence of this layer keeps the surface of the metal from sticking to the tool. The thickness of the lubricating layer formed at the deformation focus increases as the viscosity of the coating increases. For this reason, even a substance which is

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chemically inactive but has a high viscosity can be a more effective coating than a substance that interacts with the surface but has a low viscosity [1]. The thickness of the coating layer increases somewhat as the surface roughness of the blank increases.

A series of new protective process coatings (PPCs) whose special characteristics distinguish them from the conventional coatings has now been developed. These special characteristics include, in particular, high protective and heat-insulating parameters, a friction coefficient that can be regulated in definite zones of the blank being stamped, and high degrees of stretching [2–3].

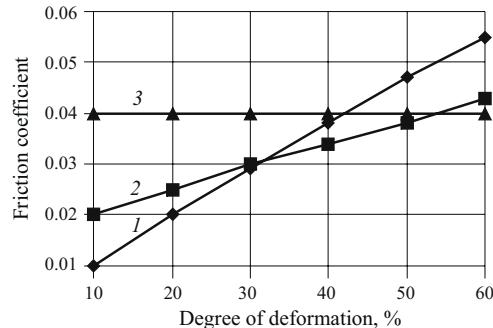
A successful solution to the problems of developing PPCs became possible when not only the coatings themselves but also the factors that reflect the interaction between coatings and the alloys being deformed and protected and the tool material at high temperatures in corrosive media were studied simultaneously.

These investigations made it possible to regulate the formation of a continuous protective coating layer and its viscosity and therefore its coefficient of friction during stamping, the degree of oxidation of the substrate as a function of the heating temperature and time for blanks and articles, the composition of a coating, the oxidation characteristics, and the technology of hot plastic working of metals and alloys.

To master resource-conserving technologies for isothermal deformation and deformation in a state of superplasticity it was necessary to determine the fundamental principles for synthesizing coatings with a qualitatively new level of properties that support high thermomechanical regimes for volume and local deformation of titanium and nickel alloys. New PPCs which combine a strong internal structure and high film-forming capacity and provide reliable protection for contact surfaces against sticking with deformation above 60% and whose contact friction coefficient can be regulated were developed.

Experiments for synthesizing coatings, investigating the variations of their physical–chemical properties, and optimizing the coating composition were planned using the simplex-grid method for heat-resistance titanium alloys in the temperature range 900–1050°C and the method of planning on a cube for high-strength alloys at temperatures 800–950°C. Mathematical models were constructed for the dependences of the properties on the composition, temperature, and deformation regimes. An electronic digital computer was used to study the property response surfaces. The optimal compositions for ÉVT35M heat-resistant titanium alloys and ÉVT88 and ÉVT88M high-strength titanium alloys were determined. The compositions of ÉVT87 and ÉVT87M coatings, which work at temperatures 800–950°C, were determined computationally from the dependences obtained.

Laser mass-spectrometry was used to monitor more effectively and investigate the gas-saturated layers of titanium alloys with PPCs. The procedure was developed to determine the local content of oxygen, nitrogen, and hydrogen in transi-



**Fig. 1.** Friction coefficient of VT22 samples with ÉVT26 coatings versus the degree of deformation: 1) glassy ÉVT26; 2 and 3) glass composition (ÉVT26 and solid phase) A and B, respectively.

tional layers up to 30–50 µm thick. This procedure made it possible to evaluate obtain qualitative and quantitative evaluations of the layers. The dependence of the oxygen content on the technology used to obtain a coating and of the effect of modifying fillers on the total oxygen content in the coatings and the transitional layers was analyzed.

The dependence of the contact friction coefficient on the degree of deformation and the composition of a coating was investigated. Glassy coatings and dispersion doped compositions with different solid-phase content were studied. These investigations showed that as the degree of deformation of glass-coated samples increases, the friction coefficient changes essentially linearly (Fig. 1). The coating is in a viscofluid state, giving a fluid friction regime. Dispersion doping of glass matrices (the introduction of a solid phase) makes it possible to increase the plasticity of a coating, stabilize the values of the contact friction, and obtain constant values of the friction coefficient in the entire range of applied pressures during hot working of blanks.

Thus, the optimal ratio of the liquid and solid modifying phases of glass compositions makes it possible to increase the plasticity of the coatings and to reach a point where during the deformation of titanium alloys the coatings spread forming a uniform, continuous, glassy film giving a constant contact friction coefficient (0.04–0.05).

A study of the oxidation and depth of the gas-saturated layer showed that PPCs effectively protect titanium and titanium alloys from oxidation at 850–1050°C. It was determined that the main factors determining the protective action of coatings are their composition and the heating temperature and composition of the alloy being protected.

In addition to decreasing the total oxygen absorption sharply, the ÉVT24, ÉVT35, and ÉVT88 coatings which were developed decrease the depth of gas-saturation of titanium alloys and decrease the oxygen concentration, which decreases the microhardness.

At 870 and 950°C the depth of the gas-saturated layer in samples of the alloy VT22 with an ÉVT24 protective coating is 2–3 times smaller than in uncoated samples. The thickness of this layer does not exceed 0.02–0.04 mm, it is easily

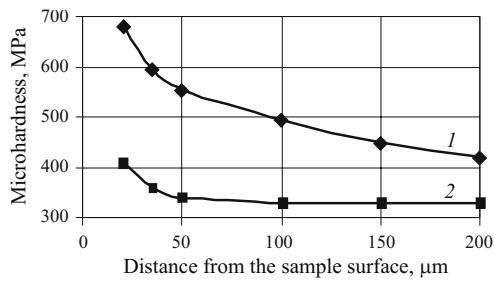


Fig. 2. Surface microhardness of VT22 titanium alloy blanks for parts of a gas-turbine engine without (1) and with (2) a coating.

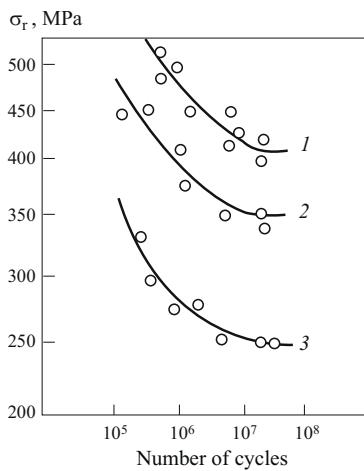


Fig. 3. Endurance limit of stamped articles (VT9 alloy) obtained with a glass-ceramic ÉVT24K coating (1), a glassy ÉVT24 coating (2), and no protection (3).

removed by sand-blasting or short-time etching, and its oxygen saturation is lower. The microhardness of samples heated with and without a protective coating is 350 MPa and 600 MPa, respectively (Fig. 2).

The use of an ÉVT24 coating during heating of VT22 alloy blanks increased the life span of the stamped articles by 30 – 40%. The VT22 alloy samples with an ÉVT24 coating

which were stamped at 950°C and had 40 and 60% deformations lasted for 15,000 and 25,000 cycles, respectively, before failure under stress 60 MPa. Unprotected samples lasted for 9000 and 13,000 cycles.

The endurance limit  $\sigma_b$  of VT9 alloy samples protected with a coating during prestamp heating and during heat-treatment increases by 40% with an ÉVT24 coating and 60% with an ÉVT24K coating (Fig. 3). The high protective parameters of an ÉVT24 coating are explained by its composition, viscosity, and wide formation temperature range.

As the solid-phase content in the glass increases, the characteristic of the removal of stamped articles from the stamping tool improves.

Pilot tests of coatings on commercial blanks confirm the experimental data obtained. Stamped articles with complex contours, unworked surface factor (USF) 0.8 – 0.9, and surface quality up to class 5 – 6 purity were made from VT22 and VT6 titanium alloys coated with ÉVT24, ÉVT88, ÉVT26, and ÉVT35 coatings (Fig. 4). The coatings made it possible to decrease the labor and energy intensiveness of manufacturing process used for the stamped articles by 30% and to increase the stability of the deforming tool by a factor of 1.5 – 2.

The ÉVT53M process coating is used to protect heat-resistant nickel and iron – nickel based alloys from oxidation and burn-out of the alloying elements during heating for stamping blanks used to make turbine blades with no allowance along the blade contour. Before stamping, blanks made of heat-resistant alloys are heated up to 1080 – 1200°C for 1.5 – 3 h. During heating without a coating the surfaces of the stamped articles oxidize and the alloying elements are burned out, which degrades the mechanical properties and surface quality. The allowance for mechanical working for removing a defective layer reaches 3 mm per side. The use of an ÉVT53M coating made it possible to decrease the labor-intensiveness of the process of fabricating articles by 30 – 35%, decrease the metal consumption by 45 – 50%, decrease the mass of the stamped article by a factor 2 – 2.5, increase the metal utilization factor from 0.2 to 0.4, decrease

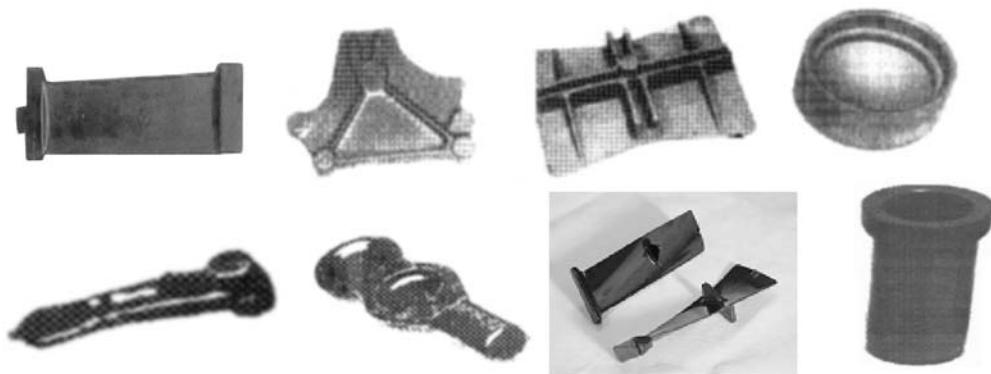


Fig. 4. Exterior view of articles stamped from titanium alloys with a protective process coating.

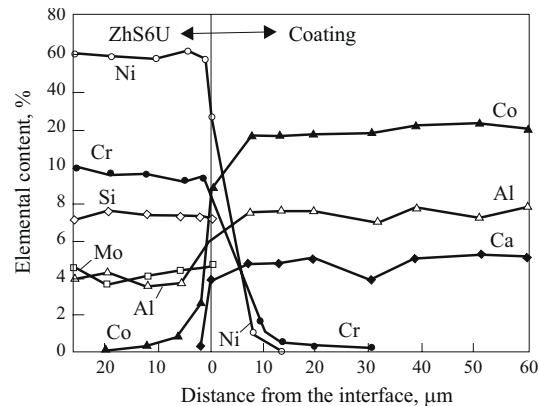
the specific pressure during stamping by 10–30%, and improve the deformation conditions.

The mechanical properties of samples, specifically, their endurance, are the chief criteria for evaluating the effectiveness of the protection provided by process coatings on heat-resistant iron–nickel alloys. Tests performed on samples of heat-resistant nickel alloys after heating showed that a coating has only a weak effect on the ultimate strength of the samples. When a coating is used, the characteristics of plasticity (elongation, contraction) with rupture of the samples are several-fold higher than for an uncoated sample.

Nickel alloys possess high strength and heat-resistance but deform with difficulty. For example, cracks often appear when ZhS6KP alloy bars are rolled. One reason for the cracking is oxidation, which causes the surface of the bars to become brittle. To improve the conditions for rolling, heated bars are wrapped with fiberglass fabric before deformation. In addition to being highly labor-intensiveness, the wrapping operation causes the bars to cool down, i.e., it promotes cracking and distortion of the rolled bars.

A study of the possibility of replacing the fiberglass fabric showed that, for example, an ÉVT100 coating reliably protects alloys from oxidation as a result of heating for rolling but it also permits decreasing the specific deformation pressure of bars by 10–12% as compared with the rolling technology which uses fiberglass fabric. An ÉVT100 protective process coating serves as a heat insulator, conserves heat in blanks, and makes intermediate heating unnecessary.

The results of x-ray spectral microanalysis of the system coating + stamping tool (heating temperature 950°C for 5 h) are displayed in Fig. 5. They show that chromium from the surface layer of the stamping tool enters the coating and silicon from the coating enters the surface of the stamping tool. The data show that the temperature stability of the coating and the stability of the stamping tool increase during hot deformation of blanks.



**Fig. 5.** Elemental distribution in the coating – stamping tool transitional layer.

The glassy and glass-ceramic protective coatings ÉVT100 and ÉVT70 were developed for hot working of corrosion-resistant VNS2, structural 30KhGSA, and high-strength VNS9 steels. The use of protective process coatings during stamping and rolling of 30KhGSA and VNS2 steel blanks protects these steels from oxidation and decarburization, increases the yield of acceptable metal up to 10%, and provides stable mechanical properties and high surface quality with geometric accuracy of the stamped articles.

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